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NATIONAL LUBRICATING GREASE INSTITUTE

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# *Report on Grease Dropping Point Methods*

By J. C. Zimmer,
N.L.G.I. Technical Committee

For a number of years both consumers and manufacturers of lubricating greases have attempted to devise a simple laboratory test that would give a reliable indication of the relative ability of greases to withstand elevated temperatures. The majority of these tests have, of course, involved estimation of temperatures at which the grease liquefies or slips off a vertical surface, such as a thermometer bulb. Attempts to improve on such dropping point tests have included measurements of the liquefaction temperature of a lump of grease floating on heated mercury and numerous types of flow point tests in which the flow through an orifice or the movement in a tube under hydrostatic pressure are employed for comparative purposes.

The results obtained from such tests frequently have not proven to be satisfactorily reproducible in different laboratories since lubricating greases do not have a true melting point and the drop or flow point is influenced by a large number of factors which cannot be readily controlled. Further, attempts to correlate the values obtained by the different test methods with service performance have not been successful.

Recognizing this condition, the American Society for Testing Materials appointed Subcommittee IV of Committee D-2 to investigate and develop a method for determining the dropping point of greases. This study extended over a period of years and a large amount of cooperative work was done before a tentative method was developed. This tentative method has recently been adopted as a standard procedure under the ASTM designation D-566. The Committee found it exceedingly difficult to develop a method which would apply to all types of greases. However, cooperative tests on the common types of lubricating greases indicate quite satisfactory reproducibility between different laboratories, as is indicated in Table I.

As far as we have been able to learn, the experience with the test procedure has been reasonably satisfactory. Certain types of products having dropping points in excess of 400°F tend to stick in the cup and char,

TABLE I

ASTM Cooperative Test
Data on Dropping Point Test (1)
A comparison of proposed tentative method and method published as information in 1937.
Temperatures in degrees Fahrenheit

| Laboratory | Cup Grease (Lime Base) New Method | Fiber Grease (Soda Base) New Method | Wheel-Bearing Grease (Soda Base) New Method | Grease (Aluminum Base) New Method |
|---|---|---|---|---|
| No. 1 | 208[a] | 355 | 443 | 241[c] |
| No. 2 | 210 | 351 | 442 | 234 |
| No. 3 | 211 | 348 | 440 | 232 |
| No. 4 | 208 | 350 | 437 | 226 |
| No. 5 | 210 | 352 | 442 | 229 |
| No. 6 | 207 | 352 | 443 | 229 |
| No. 7 | 210 | 356 | ..... | 235 |
| No. 8 | 211 | 350 | 445 | 219 |
| No. 9 | 208 | 349 | 407[b] | 225 |
| Average | 209 | 351 | 442 | 230 |
| Maximum Deviation from Average | 2(3) | 5(8) | 5 | 11(14) |
| Average Deviation from Average | 1 | 2 | 2 | 5 |

a. The data in this tabulation represent in most cases the average of 2 to 4 tests. In cases where the "maximum deviation from the average" of individual test results not shown in the tabulation exceeds the maximum deviation based upon laboratory averages, this value is shown in parentheses.

b. Data omitted from averages.

c. In this tabulation there appears to be a change of dropping point with time. Tests by Laboratories Nos. 2 to 7 were made about 10 days, and tests by Laboratories Nos. 8 and 9 about 20 days, after the test by Laboratory No. 1. A second test by Laboratory No. 1 30 days after the first test gave a value of 220°F.

(1) From American Society for Testing Materials, Proceedings of the Forty-Third Annual Meeting, Vol. 40, 1940.

and consequently show a wide range of values or no dropping point at all. Difficulty is also experienced in placing very fibrous soda base grease in the test cup, and it has been suggested that means be provided to exactly control the distance the thermometer can be inserted in the grease cup to insure greater reproducibility. The use of metal, asbestos composition, or other material of greater mechanical strength to replace the cork stopper and guide would be an improvement.

The definition of the dropping point test method included in the procedure is as follows: "The ASTM dropping point is the temperature at which the grease passes from the semi-solid to a liquid state under the conditions of the test. It should not be considered as having any bearing upon service performance." The dropping point value, however, has frequently been given far more significance than was originally planned or permitted by this definition. Obviously a grease will not be suitable at temperatures above the dropping point in applications where a lubricant of solid consistency is required. The dropping point, however, gives no reliable indication of the condition of the grease at lower temperatures where changes of sufficient magnitude to markedly affect service performance are encountered with many types of greases. For example, conventional lime soap greases of firm consistency have ASTM dropping points of 200°F or higher, but they are seldom if ever recommended for service where the temperature runs consistently above 160°F. The spread between dropping point and a safe service temperature maximum frequently exceeds 100°F with soda and mixed base products.

In view of this experience it appears that the ASTM dropping point can at best be used only as a very qualitative indication of the resistance of greases to heat, for example, to distinguish conventional lime base cup grease from high melting point types, such as soda, etc. Thus, an ASTM dropping point minimum might be employed by consumers to exclude certain types of greases. There also is a tendency to employ the dropping point as a routine control test for uniformity on grease purchases from given source. However, since the dropping point is usually not a reliable index of soap content, nor subject to as satisfactory control during manufacture, as is the worked penetration, the latter should serve as a more reliable index of uniformity.

The failure of the ASTM dropping point to give a reliable indication of the maximum service temperature or to exclude all products which might prove unsatisfactory in a given application by the ASTM dropping point has not resulted in a decline in the use of this type of laboratory test. On the contrary, a number of the older type tests which it was hoped would be superseded by the ASTM have continued in use and a number of new modifications have been developed with resultant confusion and undue burden of numerous test procedures on suppliers. Results reported by Mr. Georgi, at the 1942 meeting, from the National Lubricating Grease Institute's Survey on Test Methods, indicate that at least the seven methods shown above are in fairly common use:

DROPPING POINTS—SODA BASE GREASES

| | 280 Penet. 120 Vis. Oil | 220 Penet. 120 Vis. Oil | 260 Penet. 60 Vis.Oil | 260 Penet. 90 Vis. Oil |
|---|---|---|---|---|
| ASTM D-566 | 295° | 320° | 305° | 330° |
| Ubbelohde | 330° | 340° | 330° | 355° |
| Spiral Wire on Bulb | 335° | 345° | 320° | 370° |
| 3/12" x ¾" Tube | 235° | 240° | 235° | 315° |
| ¼" x ½" Tube | 225° | 250° | 220° | 260° |
| Wire Thimble | 220° | 255° | 230° | 280° |
| Glass Tube (5 x 25 mm.) | 240° | 280° | 200° | 240° |

It will be noted that a very wide range of values was obtained by these methods. Certain of the methods involving the use of a small amount of the test sample on a good support give quite high values, while others, requiring a larger sample, which is free to flow out of a relatively large tube give values as much as a hundred degrees lower. These latter values appear to approximate more nearly the maximum service temperatures, and suggest that measurement of flow temperature or resistance to flow over a range of temperatures might be a reliable criterion of performance characteristics at elevated temperatures.

Several test methods involving flow temperatures or rates of flow have been suggested, and of these a preliminary investigation has been made of the so-called "aluminum block test," proposed by Mr. Moir, of the Pure Oil Company. As shown in Figure A, the test apparatus consists of a 6½" high, 12½" long, and 2⅜" thick aluminum block, internally heated by electrical resistance bars, and having the ⅛" x 4" channels connected to a ⅛" x ½" circular reservoir cut in the face of the block. The test sample is placed in the reservoir and smoothed off flush with the block surface. The temperature of the block is then raised at a uniform rate. The temperature at which any oil separates from the grease and runs down the channel is noted. Also, the temperature of initial flow of the grease,

TABLE II

Comparison of Aluminum Block Test
With Dropping Point Determination D 566

| % Soap | Mineral Oil Vis/100°F | Worked Penetration | D 566 Dropping Point °F | Aluminum Block Test Temperatures: Separation of Oil | First Movement of Grease | 4" Flow of Grease |
|---|---|---|---|---|---|---|
| Calcium Soap Greases | | | | | | |
| 14 | 100 | 290 | 195 | ..... | 154 | 158 |
| 12 | 300 | 310 | 200 | ..... | 195 | 197 |
| Aluminum Base Greases | | | | | | |
| 10 | 300 | 290 | 210 | ..... | 205 | 240 |
| 18.5 | 475 | 170 | 320 | ..... | 235 | 320 |

TABLE III

Comparison of Aluminum Block Test
With Dropping Point Determination D 566

| % Soap | Mineral Oil Vis/100°F | Worked Penetration | D 566 Dropping Point °F | Aluminum Block Test Temperatures: Separation of Oil | First Movement of Grease | 4" Flow of Grease |
|---|---|---|---|---|---|---|
| Lithium Base Grease | | | | | | |
| 13.5 | 60 | 285 | 382 | ..... | 392 | 392 |
| Sodium Soap Greases | | | | | | |
| 18.5 | 290 | 300 | 340 | 239 | 275 | 280 |
| 15.0 | 290 | 251 | 347 | 324 | 334 | 350 |
| Mixed Base Greases | | | | | | |
| 18.5 | 200 | 270 | 340 | 197 | 250 | 328 |
| 19.0 | 330 | 225 | 347 | 356 | 370 | 373 |

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FIG. A. *Aluminum Block Test Apparatus*

or the temperature at which the grease has moved a given distance down the channel, is reported. Test results obtained by this method are compared with the ASTM dropping points in Tables II and III.

This test procedure permits observation of any tendency to separate an appreciable amount of oil at temperatures appreciably below the dropping point and also shows that some products will flow under their own weight at temperatures considerably below the ASTM dropping point. Correlation with service performance will be necessary to evaluate the significance of the test results. However, since the flow temperature in a number of cases equals or exceeds the dropping point, it is doubtful that the test would satisfactorily predict the high temperature performance characteristics of some types of greases.

Consideration of the shearing and hydrodynamic forces set up in a laboratory journal or anti-friction bearing leads to the conclusion that a knowledge of the resistance to flow of a grease under loads greater than gravity should be of considerable aid in predicting performance. Grease lubrication failures at elevated temperatures probably are due mainly to two causes, oxidation and lacquer formation which result in a cold stuck bearing, or failure of the grease to retain its solid consistency, stay in the bear-

*(Continued on Page 6)*

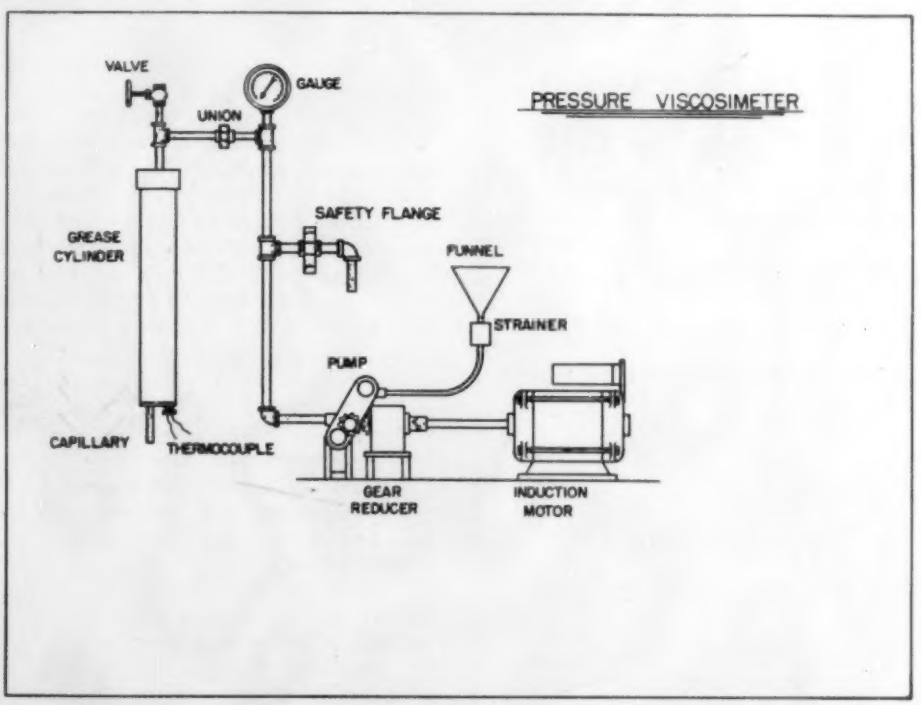


FIG. B

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# *Grease Production and Fat-Fatty Acid Consumption by the Lubricating Grease Industry*

By SYDNEY BEVIN* AND C. W. GEORGI**

The revocation of Petroleum Administrative Order No. 10, under date of October 27, 1943, occasioned no noticeable expressions of regret by manufacturers of lubricating greases. Many manufacturers had reported difficulties in complying with Order No. 10, which may be summarized as follows:

(1) Decreased grease poundage output per Kettle due to generally lower yields from fatty acid soaps as compared to soaps of whole fats; longer kettle processing time per batch due to inexperience of many greasemakers with fatty acids; higher percentage of batch rejections and batch reworkings.

(2) Difficulties with storage and handling equipment for fatty acids in those plants which had not set up prior to the war special non-corrodable tankage and piping systems.

(3) Higher costs of greases, first because of the higher cost of fatty acids and second, because of the generally lower grease yields per pound of fatty acid soap as compared to whole fat soaps.

(4) Difficulty in securing supplies of fatty acids of uniform and adequate quality.

However, in accord with the old saying that it is an ill wind, indeed, that blows no good, Order No. 10 did accomplish one very useful result. The quarterly reports on lubricant production and fat-fatty acid consumption, as required from manufacturers by Order No. 10, have provided for the first time accurate and comprehensive information concerning the size of the grease industry. Through the courtesy of the Refining Division of the Petroleum Administration for War these data have been supplied to the N.L.G.I. and have been released for publication. Table I lists the data by quarters for the first three quarters of 1943 and of 1941.

It should be noted that the figures for the first quarters of 1943 and 1941 are from the reports of grease manufacturers in the U. S. who produced at the rate of not less than 1,000 Tons (2,000,000 lbs.) of lubricating greases per year and who were required to report under the original P.A.W. Recommendation No. 58. The figures for the second and third quarters of '43 and '41 are from the reports of grease manufacturers who produced at the rate of not less than 500 Tons (1,000,000 lbs.) of greases per year, and who were required to file quarterly reports under P.A. Order No. 10. The 2nd and 3rd quarter data thus include all but the very small manufacturers and it seems reasonable to assume that at least 90% of the industry on the basis of output is included.

On the basis of the 2nd and 3rd quarter 1943 data, the current output of lubricating greases in the United States exceeds 500,000,000 pounds per year and the consumption of fats and fatty acids runs from 60 to 65 million pounds annually.

Aside from the production and fat consumption figures, the data in Table I provide other interesting information. The average total fatty content of lubricating greases is very close to 12%. In fact it is quite remarkable how uniform the fatty content averages over the three quarter periods of 1943 and 1941 respectively.

It is also noteworthy that in normal operation, as per the 1941 figures, the grease industry consumes considerable quantities of fatty acids. Although the majority of calcium and soda base greases are made from whole fats as a matter of preference and sometimes necessity, many special industrial greases and practically all aluminum base greases are made from fatty acids.

In 1941 approximately 30% of the total fatty materials used for grease making were fatty acids and in 1943, under the requirements of P.A. Order No. 10, this figure rose to 45%. With the suspension of Order No. 10, it is most probable that the old ratio will return, so that the current consumption of fatty materials by the grease industry may be estimated as approximately 18 to 20 million pounds of fatty acids and 40 to 45 million pounds of whole fats annually.

Since the purpose of P.A.W. Recommendation No. 58 and P.A. Order No. 10 was to increase supplies of glycerine by requiring increased use of fatty acids as replacement for whole fats, the data in Table II are of interest.

Based on the 2nd and 3rd quarter figures, P.A.W. Order No. 10 effected an increased use of fatty acids, equivalent to approximately 10 to 12 million pounds per year, with a resultant theoretical conservation of glycerine amounting to about one million pounds annually as a maximum.

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*Sydney Bevin, Chief Engineer, Fiske Bros. Refining Company, Chairman, Sub-Committee on Manufacturing Problems, N.L.G.I.

**C. W. Georgi, Technical Director, Quaker State Oil Refining Corporation, Chairman, N.L.G.I. Technical Committee.

It would appear that the lubricating grease industry is not a major consumer of glycerine or conversely an important source of glycerine conservation. This is particularly the case when consideration is given to the fact that essentially 100% of the lubricating greases manufactured reach vital and essential uses in the fields of transportation, industry, agriculture and military.

TABLE I

Lubricating Grease Production and Fat-Fatty Acid Consumption

| | | 1943 | 1941 |
|---|---|---|---|
| (1) Total Grease Manufactured in U. S. | 1st Quarter | 85,545,000 lbs. | 72,912,000 lbs. |
| | 2nd Quarter | 124,990,000 lbs. | 109,857,000 lbs. |
| | 3rd Quarter | 129,256,000 lbs. | 119,524,000 lbs. |
| (2) Total Fats Used | 1st Quarter | 5,930,000 lbs. | 6,257,000 lbs. |
| | 2nd Quarter | 8,178,000 lbs. | 9,742,000 lbs. |
| | 3rd Quarter | 8,135,000 lbs. | 10,473,000 lbs. |
| (3) Average % Fat in Grease Produced | 1st Quarter | 6.93% | 8.58% |
| | 2nd Quarter | 6.54% | 8.87% |
| | 3rd Quarter | 6.29% | 8.76% |
| (4) Total Fatty Acids Used. | 1st Quarter | 5,124,000 lbs. | 3,146,000 lbs. |
| | 2nd Quarter | 6,732,000 lbs. | 3,702,000 lbs. |
| | 3rd Quarter | 7,164,000 lbs. | 4,526,000 lbs. |
| (5) Average % Fatty Acids in Grease Produced | 1st Quarter | 6.0% | 4.31% |
| | 2nd Quarter | 5.39% | 3.37% |
| | 3rd Quarter | 5.54% | 3.79% |
| (6) Average % Fats and Fatty Acids in Grease Produced. | 1st Quarter | 12.93% | 12.89% |
| | 2nd Quarter | 11.93% | 12.24% |
| | 3rd Quarter | 11.83% | 12.55% |

TABLE II

Glycerine Conservation Effected by Petroleum Administrative Order No. 10

| | 3rd Quarter 1943 vs. 1941 | 2nd Quarter 1943 vs. 1941 | 1st Quarter 1943 vs. 1941 |
|---|---|---|---|
| (a) Actual Decrease in Fat Consumption | 2,338,000 lbs. | 1,564,000 lbs. | 327,000 lbs. |
| (b) Decrease in Fat Consumption corrected for larger production in 1943 | 3,188,000 lbs. | 2,912,000 lbs. | 1,410,000 lbs. |
| (c) Actual Increase in Fatty Acid Consumption | 2,638,000 lbs. | 3,030,000 lbs. | 1,978,000 lbs. |
| (d) Increase in Fatty Acid Consumption corrected for larger production in 1943 | 2,265,000 lbs. | 2,520,000 lbs. | 1,437,000 lbs. |
| (e) Theoretical Glycerine Saving, Based on 10% yield from Fatty acids per line (d). | 226,000 lbs. | 252,000 lbs. | 144,000 lbs. |

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## *Report on Grease Dropping Point Methods*

*(Continued from Page 3)*

ing and provide lubrication. Conventional lime soap cup greases, for example, will melt and run out of a bearing at temperatures above 210°F, leaving a dry soap residue, or no lubricant at all, either of which will result in bearing failure. Greases may also become quite soft and flow readily under very low stress at temperatures appreciably below their dropping point. Such a product might leak out of a bearing which is not equipped with oil-tight seals, or the churning action of the bearing could force it out, resulting in lubrication failure. A number of examples where the relative resistance to flow or apparent viscosity could be correlated with service performance were included in the paper on, "The Flow Characteristics of Lubricating Greases," presented at the 1942 Meeting of the National Lubricating Grease Institute. These examples, however, were limited to ambient and sub-zero temperature measurements. During the past year we have modified an SOD Pressure Viscosimeter to permit measurements over a range of elevated temperatures, in an effort to secure information which might predict high temperature performance.

Figure B is a sketch of the original SOD Pressure Viscosimeter with motor, gear box, pump, cylinder and capillary in place. The Zenith pump, having a constant volume displacement per revolution, is the essential part of the instrument and for equilibrium pressure the hydraulic oil displacement by the pump forces an equivalent volume of the grease through the capillary. Knowing this flow rate and the dimensions of the capillary, only the pressure need be observed to calculate apparent viscosities from Poiseuille's equation.

Our apparatus for viscosity studies at high temperatures involves the enclosure of the cylinder and capillary in an air bath or oven (Figure C). This oven, which is insulated, contains a fan for circulating air and a series of strip heaters with a "variac" resistance and transformer control in the heater circuit. The Zenith pump is started and the grease is extruded at room temperature until equilibrium pressure is reached at the desired flow rate. The oven is then heated under controlled conditions to give 0.8 ± 0.1°F/min. temperature rise in the grease at the point of efflux to the capillary. Both the temperature and pressure are recorded continuously. A relatively slow flow rate of about 0.01 cc./sec. or 100 reciprocal seconds was chosen since this should approximate conditions at which leakage from a bearing might be expected. Also, it permits a determination over the temperature range of 100°F to 350°F on one grease cylinder charge. The capillary used has a length of 6.56 cm. and a radius of 0.0503 cm. This gives readily determinable pressures for cup greases and the various types of high temperature anti-friction bearing greases. The data are then plotted, pressure versus temperature, and since, under the conditions of the test, viscosity is proportional to the pressure required to cause flow, the resulting plots are essentially viscosity-temperature diagrams.

A number of greases have been tested in this manner and the data for some of them are shown in the accompanying Figures. Grease A and Grease D (Figure 1) are ordinary lime soap cup greases containing small amounts of water as a stabilizing or soap dispersing agent. These products, of course, liquefy in the neighborhood of 200°F, as indicated by the dropping point data. However, it should be noted that there is a rapid drop off in the pressure or viscosity at 160° to 170°F, which is usually considered the maximum satisfactory service temperature for a product of this type. Materials having higher boiling points than water are occasionally used as stabilizing or modifying agents in lime soap greases to produce the so-called high melting point or reversible lime soap greases. Grease B and Grease C are cited as examples of such products, and it will be noted the viscosity remains appreciable even above 300°F. Although both Grease B and Grease C have about the same viscosity at 325°F, the indication is that Grease C would be a more reliable lubricant below 300°F, particularly in the range of 200 to 250°F where it has three times the viscosity of Grease B, and thus would be less apt to work out of a bearing. There appears to be good agreement between the drop point and the temperature at which very low viscosities (1.0 lbs./in.² pressure) are approached with the conventional lime soap cup greases. Grease C, however, shows this minimum pressure at 340°F in comparison to a drop point in excess of 400°F.

(To Be Continued)

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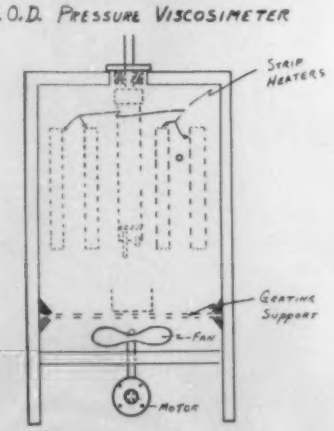


FIG. C.

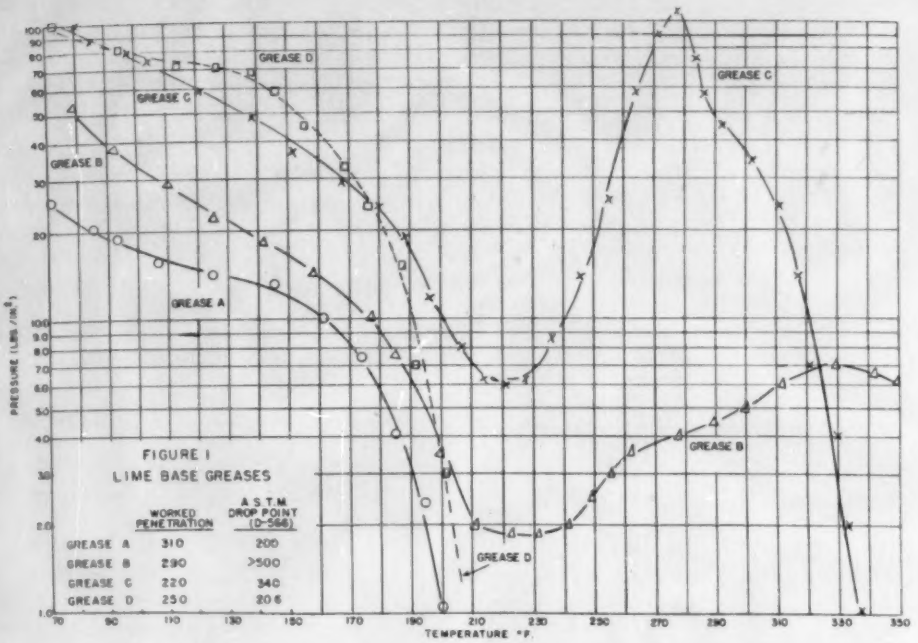


FIGURE I
LIME BASE GREASES

| | WORKED PENETRATION | A.S.T.M. DROP POINT (D-566) |
|---|---|---|
| GREASE A | 310 | 200 |
| GREASE B | 290 | >500 |
| GREASE C | 220 | 340 |
| GREASE D | 250 | 206 |

# *Containers*

As reported by W. H. Oldacre, Chairman
Container Committee

It is somewhat difficult to adequately appraise the container situation in the petroleum industry at the present time. Sheet for containers is still extremely limited in quantity and the industry must do everything possible to conserve containers. The drum situation is critical, as a very meager quantity of drum sheet is available for domestic civilian needs.

An amendment to Order M-81, dated October 23, permits the use of 1-pound and 5-pound sizes of grease cans in black plate in addition to the 10-pound and 25-pound sizes previously allowed. A can for these capacities can be made reasonably tight with a friction top cover, and will undoubtedly be extensively used.

Under the new Order L-197, dated October 2, a new procedure of making application for steel drums is inaugurated. This procedure involves a simplified Form 3233, which serves a three-fold purpose. If the product to be packed is on the restricted list, the Form is considered first as an appeal. If the appeal is cleared, it is processed as a request for the purchase of drums. After the purchase is authorized, then the rating under which they may be bought is assigned. There should be little delay as the procedure has been further streamlined by an arrangement with the WPB whereby the Forms may be sent by petroleum marketers direct to PAW. Applicants should get their applications back within a few days.

There has been some agitation among marketers for a more liberal application of restrictions on the use of 25-pound or 5-gallon pails. At the present time it seems that such agitation serves no useful purpose and may result in the tightening up of certain favorable rulings under which we have been operating.

Due to a scarcity of paper pulp certain restrictions have been placed on the manufacture of fiber containers. As a great deal of effort has been expended in developing this industry so that substitute packages will be available in case steel packages became scarcer, it is felt that our industry should use fiber containers wherever possible to cooperate with the industry in order that recent gains may be preserved.

The Container Division of the War Production Board has recently developed a program emphasizing the re-use of containers of all kinds in which our industry should enthusiastically participate. Referring to drums, the WPB conservation program emphasizes the critical scarcity and makes the following recommendations as an aid to conservation:

1. Do not drop!
2. Use a little oil to avoid stripping threads in removing plugs.
3. Replace plugs carefully when drum is empty.
4. Keep drums under cover when emptied.

We know that our industry is willing and anxious to co-operate in this program to help the nation and ourselves.

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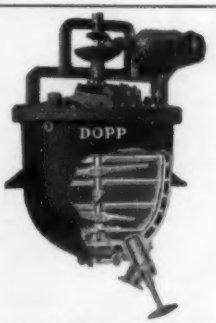


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